

MANUFACTURE AND ANALYSIS OF MgB_2 SUPERCONDUCTING WIRE WITH ADDITION CARBON NANO TUBE (CNT) BY IN-SITU

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ABSTRACT

The study has been conducted on making samples of MgB_2 superconducting wire, by adding 1% CNT with situ method. MgB_2 is a superconducting material that has a critical temperature of 39 K. The making process begins with weighing Mg powder, B powder, and CNT powder. After weighing, a grinding process with mortar agate is conducted for 45 minutes. The grinding results are inserted into the SS304 tube, and then the rolling wire process is carried out. Afterwards, the wire is cut and had sintering process with temperature variations of 750 °C, 800 °C, and 850 °C. Characterization is carried out using XRD to determine the phase formed, SEM to see the morphology, and Cryogenic Magnet to determine the nature of superconductivity. The study finds that the critical temperature produced in the sample of MgB_2 wire without the addition of CNT is 33.77 K, while in the sample MgB_2 wire with the addition of 1% CNT is 36.67 K. Thus, the addition of 1% CNT increased the critical temperature value.

KEYWORDS: MgB_2 , CNT, SS304, Sintering, Rolling Wire & Cryogenic Magnet

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1. INTRODUCTION

The study on superconductors, which are growing nowadays has made many researchers around the world continue to improve the quality of superconducting compounds. The researchers are trying to find superconducting materials which have a critical temperature (T_c) equal to room temperature. One of these superconducting materials is MgB_2 . The development of MgB_2 material which has a critical temperature (T_c) 39 K was found in January 2001. The specialty of this superconductor is that, it has a fairly high critical temperature, simple crystal structure, high coherence length, high critical current density, high critical field (40 T) and grain boundary transparency which make MgB_2 a good material for large-scale applications, namely electronic devices.

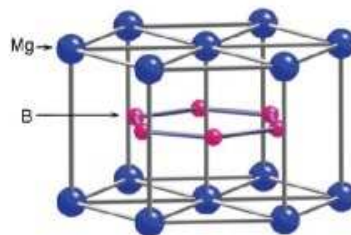


Figure 1: Crystal Structure of MgB_2

The research and the development of MgB_2 material are carried out in order to replace Nb_3Sn material. MgB_2 , besides having a higher critical temperature value, is cheaper than Nb_3Sn .

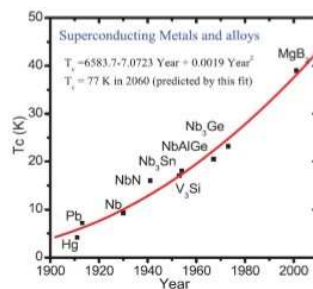


Figure 2: Critical Temperature (T_c) of Several Metal and Alloy Materials

Superconductors are materials that have zero electrical resistance at very low temperatures. Superconductors can even conduct electrical current without a voltage source. The characteristics of superconducting materials are the magnetic fields in superconductors worth zero and experiencing the meissner effect. The resistivity of a material is zero, if it is below its critical temperature.

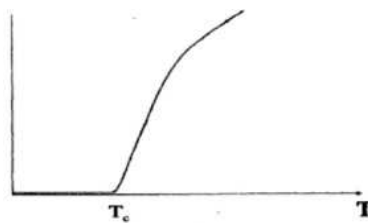


Figure 3: Graph of the Relationship between Resistivity and Temperature

When an electric field is applied to the material, the electrons will get accelerated. The electric field will release electrons in all directions and pound the atoms in the lattice. This cause makes the electrical resistance of the conductor metal.

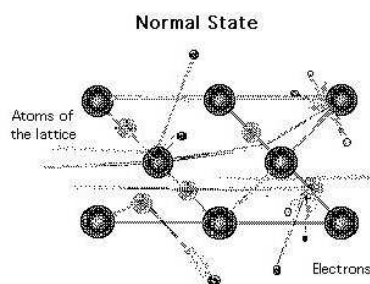


Figure 4: Atomic Normal State of Lattice on Metal

In superconducting materials, there is also an interaction between electrons and atomic nuclei. But, electrons can pass through the nucleus without experiencing the resistance of the lattice atom. If there are two electrons passing through the lattice, the second electron will approach the first electron because the attraction of the nucleus of the lattice atoms is greater. This style exceeds the repulsive force between electrons so that the two electrons move in pairs.

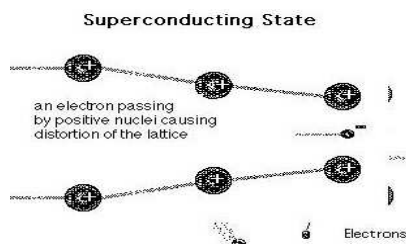


Figure 5: State of Atomic Superconductor Lattice on Metal

Another characteristic of superconductors is perfect diamagnetism. If a superconductor is placed in a magnetic field, there will be no magnetic field in the superconductor. This happens because the superconductor produces a magnetic field in the material that is in the opposite direction to the external magnetic field given. The same effect can be observed, if the magnetic field given to the material in normal temperature is then cooled to become a superconductor. At critical temperatures, the magnetic field will be rejected. This effect is called as the Meissner Effect.

2. EXPERIMENTAL METHOD

Wire MgB_2 samples were prepared by in situ powder-in-tube (PIT) method. For the tube, we use SS 304 tubes of 9 cm long with an outside diameter (OD) of 7 mm and inside diameter (ID) of 5 mm. Commercially available magnesium powder and amorphous boron powder and 1% Carbon Nano Tube (CNT) was mixed. Powders were mixed and ground with mortar agate and pestle air for about 40 minute to get homogeneous fine powder. Then, we inserted the powder into the open end of the tube. Both ends of the tubes were sealed by SS 304 plugs. The filled tubes were groove rolled into square wires with lengths of 42 cm.

Samples were then cut until every sample has length of 7 cm, and then we put it into SS304 tube to prevent oxidation at the end of the tube for about 15 minute. After that we sintered the samples at (750°C , 800°C and 850°C) for 120 minute, then we left it to reach room temperature in the furnace. We removed the samples from the SS 304 tube and the reacted MgB_2 wire was obtained. The phase composition of sintered samples was examined by X-ray diffraction (XRD) using XRD PHILIPS Panalytical Empyrean PW1710. The temperature dependence of resistivity in the sintered samples was measured by a DC four-probe method using Cryogenic Magnetic Cryotron FR Oxford machine, and to check the microstructure of the samples, we used SEM-EDX JSM-6390AJEOL

3. RESULTS AND DISCUSSIONS

Figure 6 and 7 show the manufacture process of MgB_2 wire samples with the addition 1% CNT. At the sintering temperature of 750°C and 800°C , the sample MgB_2 phase is formed, but Figure 8 shows the manufacture process of MgB_2 wire samples with the addition 1% CNT. At the sintering temperature of 850°C , the sample does not form MgB_2 phase. The addition of CNT with high temperature makes Mg and B hard to react. The Mg, which acts with O causes the formation of MgO phase. The MgO phase also appears because, the sintering process is carried out at high temperatures over a long period of time (± 120 minutes), considering that the Fe tube is not yet fully vacuumed. The peak of CNT is undetectable due to C atoms in the CNT being *amorphous*.

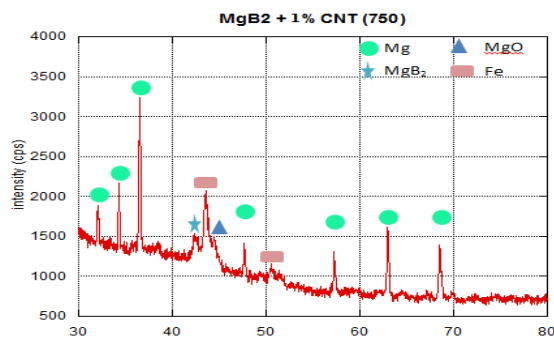


Figure 6: Diffraction Pattern of MgB₂ Wires with Sintering Temperature 750°C

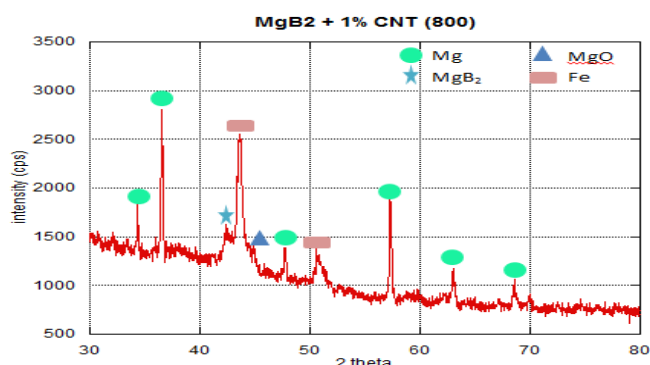


Figure 7: Diffraction Pattern of MgB₂ Wires with Sintering Temperature 800°C

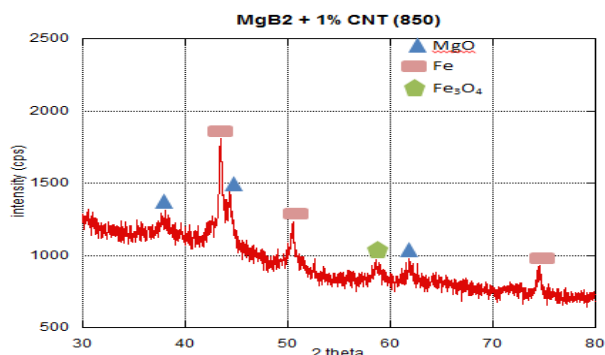


Figure 8: Diffraction Pattern of MgB₂ Wires with Sintering Temperature 850°C

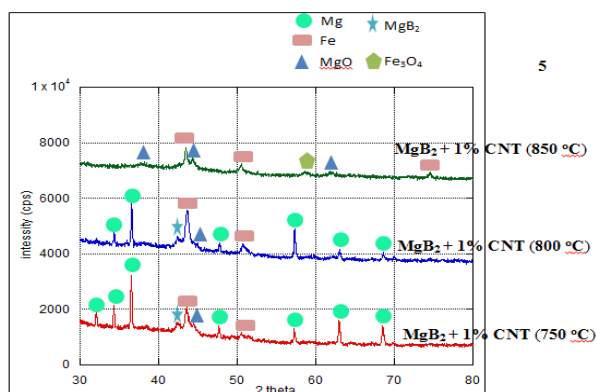


Figure 9: Combined XRD Patterns

Figure 10 shows some photos of the surface morphology of MgB_2 wire samples with the addition of CNT, synthesized by the *in situ* method. In figure 12, the surface morphology of the sample is not properly agglomerated, so it forms small grains and it doesn't bind to one another.

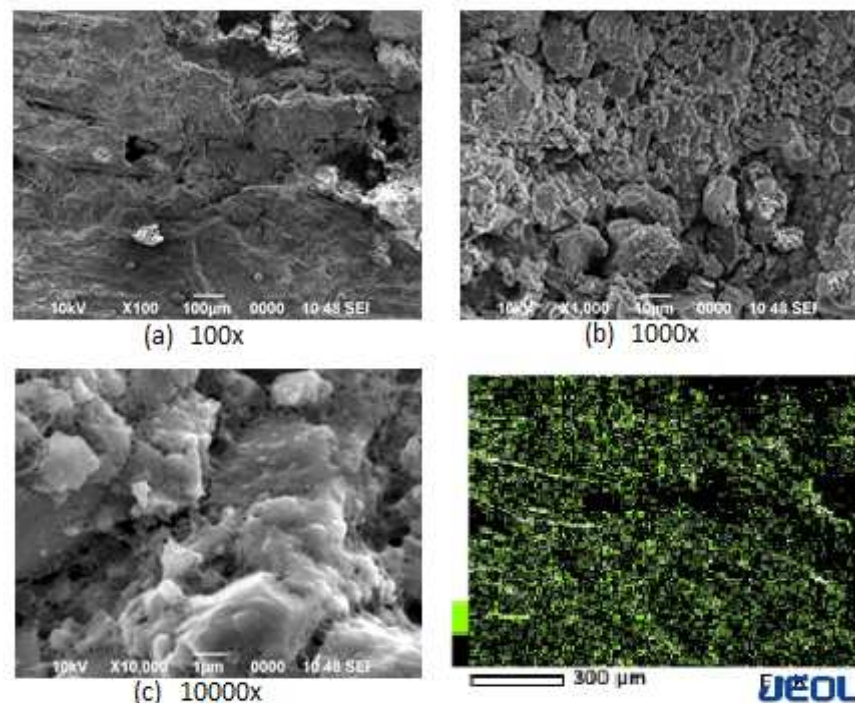


Figure 10: The Microstructure of MgB_2 Wire from Front View with Temperature Sintering $750^{\circ}C$

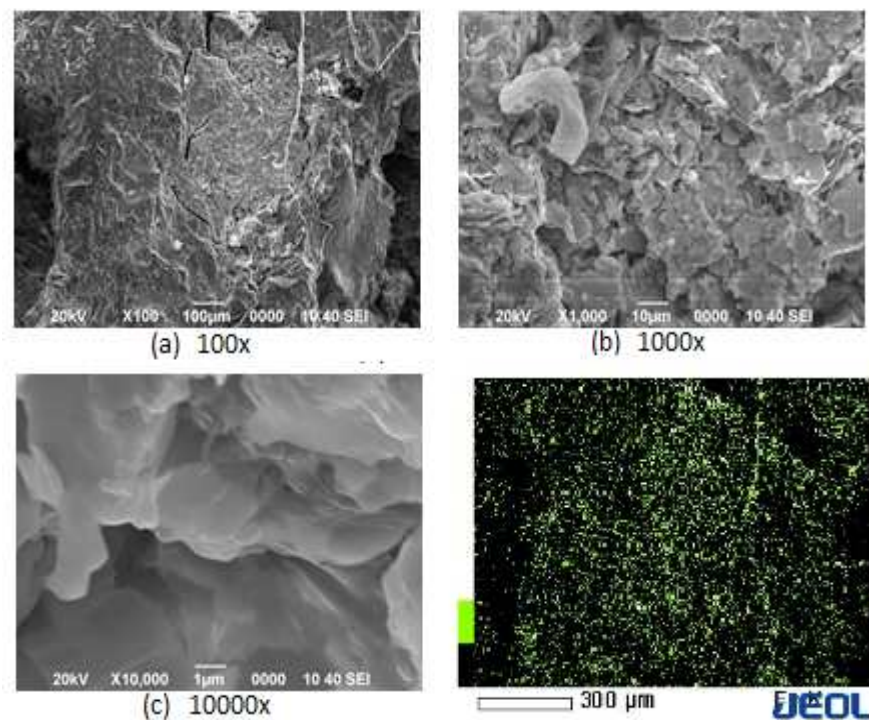


Figure 11: The Microstructure of MgB_2 Wire from Front View with Temperature Sintering $800^{\circ}C$

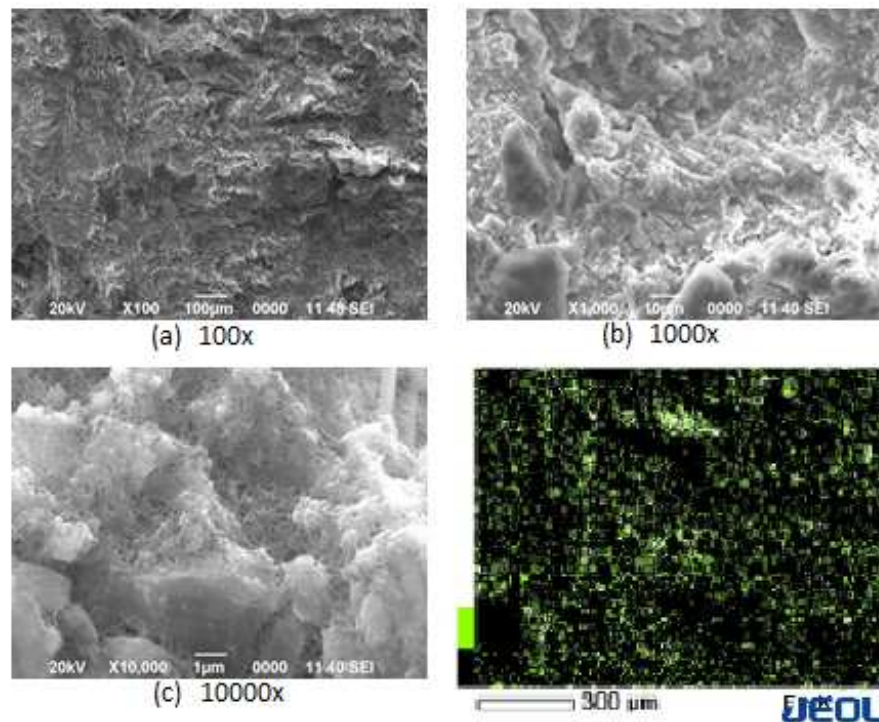


Figure 12: The Microstructure of MgB₂ Wire from Front View with Temperature Sintering 850⁰C

In Figure 12, the surface morphology of the MgB₂ wire sample shows a considerable porosity. Figure 13 is the result of identification of MgB₂ samples with the addition of CNT and variations in sintering temperature (750; 800; 850) ⁰C. Sample **a** has superconducting properties, which can be seen from the curve that shows, the resistivity decreases very drastically to 0. It also has T_{conset} of 42.75 K and T_{c0} = 33.77 K. Furthermore, sample **b** also shows superconductor properties. Its curve shows the decrease in resistivity drastically to 0, having T_{conset} of 43.17 K and T_{c0} of 36.67 K, whereas in sample **e**, the sample does not show superconducting properties, it appears that the curve doesn't show a decrease in resistivity. This is because, the diffusion reaction of MgB₂ with imperfect CNTs with the emergence of new phases or impurity phases and high sintering temperatures can damage the superconducting properties of MgB₂ material.

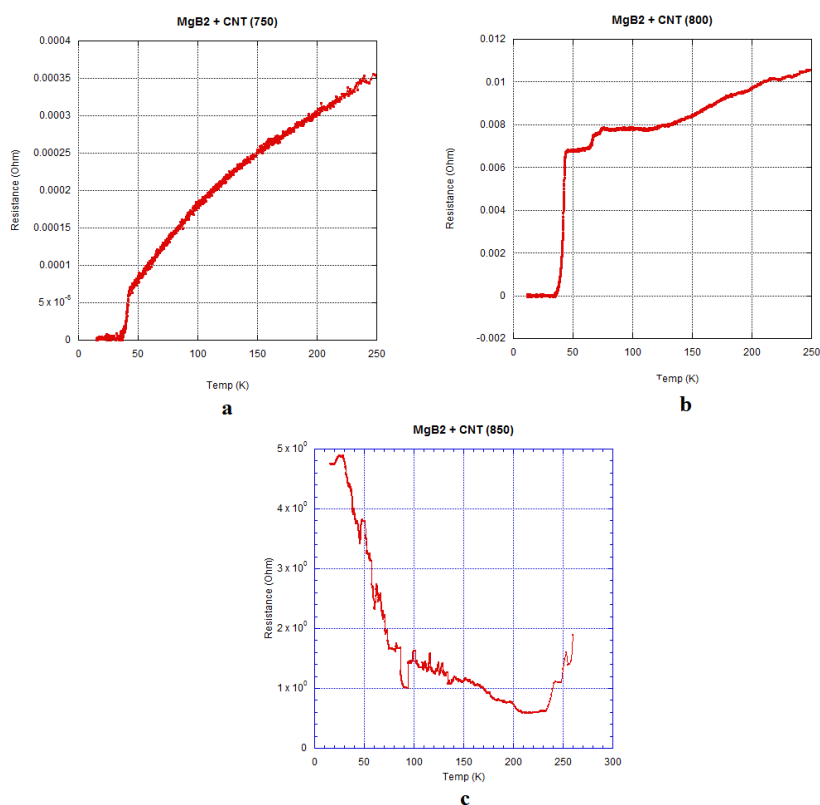


Figure 13: The Resistivity Curve against Critical Temperature (T_c)

Table 1: The Data of Critical Temperature in Each Sample

Sample	Suhu ($^{\circ}\text{C}$)	$T_{c_{\text{onset}}}$ (K)	T_{c_0} (K)	ΔT_c (K)
$\text{MgB}_2 + 1\% \text{ CNT}$	750	43.17	36.67	6.50
$\text{MgB}_2 + 1\% \text{ CNT}$	800	43.64	35.29	8.35
$\text{MgB}_2 + 1\% \text{ CNT}$	850	-	-	-

4. CONCLUSIONS

From the study above, it can be concluded that:

- The manufacture process of the wire sample has successfully reduced 33% -50% or from $\varnothing 6$ mm to $\varnothing 4$ - $\varnothing 3$ mm. Visually, the sample had almost no defects, only slightly fine cracks.
- The sintering temperature is optimal at 750°C . With the addition of CNT, the temperature value of $T_{c_{\text{onset}}}$ increases compared to the sample without the addition of CNT.
- T_c is formed at a temperature of 750°C - 800°C . At 850°C T_c is not formed, which indicates that the nature of the superconductor is not formed.

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